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EFFECT OF TEMPERATURE AND OXYGEN ON THE STRENGTH OF ELASTOMERS

by

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Akron, Ohio, 44325-0301

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## 20. ABSTRACT continued

of 20 to 32 kcal/mole were deduced for the tear strength, much larger than predicted by the WLF relation for changes in viscous losses with temperature far above  $T_g$ . Another temperature-dependent term in the tear strength is therefore inferred. It is suggested that threshold strength,  $G_0$ , measured in the absence of viscous effects, is itself temperature-dependent. Changes in  $G_0$  by factors of between 2X and 5X as the temperature is reduced from 130° to 25°C would account for the observed discrepancies. Tear rates were also measured under either steady loading or intermittently-applied loads as a function of atmospheric pressure, over the range of 0.1 to 760 mm Hg. They were found to decrease to about 25-30% of the values at one atmosphere, on removing air almost completely. This effect of air pressure is attributed to attack of oxygen (in air) on highly-stressed bonds, either sulfidic or hydrocarbon in nature, accelerating the tear process.

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# Effect of Temperature and Oxygen on the Strength of Elastomers

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## 1. Introduction

Crack growth occurs in rubber vulcanizates under stress when a sufficient amount of elastically-stored energy  $\underline{W}$  is released by growth of the crack. The criterion is (1):

$$-(\partial W / \partial S)_d \geq G_c \quad (1)$$

where  $\underline{S}$  is the area torn through by the crack and  $\underline{G}_c$  is the energy required for tearing through unit area ( $\text{J/m}^2$ ), a measure of strength. The derivative on the left-hand side of Equation 1 is taken under conditions of constant displacement  $\underline{d}$ , to avoid the complexity of further work input as the crack grows.

It is found, in practice, that the value of  $\underline{G}_c$  is not well-defined. Cracks grow to some extent when the available energy  $\underline{G}$ , defined by the left-hand side of Equation 1, exceeds a threshold level  $\underline{G}_0$ , of the order of  $50 \text{ J/m}^2$  (2,3). The amount of crack growth then depends upon the extent to which  $\underline{G}$  exceeds the threshold level. It is only at a much higher level, denoted here  $\underline{G}_c$ , of the order of  $10,000 \text{ J/m}^2$ , that catastrophic tearing occurs.

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<sup>1</sup>On leave of absence from Bridgestone Corporation, Japan.

The slow or small-scale tearing that takes place for stresses corresponding to values of  $\underline{G}$  between these two limits,  $\underline{G}_0$  and  $\underline{G}_c$ , is referred to as mechanical fatigue cracking. It is more pronounced under intermittently-applied loads, but for non-crystallizing elastomers, such as SBR, steady tearing takes place if the load is maintained continuously, at a slower rate. For a wide range of applied loads and tearing energies  $\underline{G}$ , the rate of crack propagation  $\underline{dc}/\underline{dt}$ , where  $\underline{t}$  is the duration of steady loading, or  $\underline{dc}/\underline{dn}$ , where  $\underline{n}$  is the number of load applications, is found to increase with  $\underline{G}$  in accordance with empirical power laws:

$$\underline{dc}/\underline{dt} = A \underline{G}^{\alpha} \quad (2)$$

or

$$\underline{dc}/\underline{dn} = A' \underline{G}^{\alpha'} \quad (3)$$

where the exponents  $\underline{\alpha}$  and  $\underline{\alpha}'$  are found to be about 4 for SBR and 5 to 6 for BR. The crack growth constants for steady tearing ( $\underline{A}$ ) and intermittent tearing ( $\underline{A}'$ ) are characteristic of the compound and the test conditions (4).

In fact, although the test conditions, notably the test temperature and the presence or absence of air, are known to have large effects on the rate of crack propagation, there is relatively little quantitative information available, especially at high

temperatures. We have therefore studied these effects in detail for three well-defined vulcanizates of SBR, having different types of crosslink, and a sulfur vulcanizate of BR. The results are reported here.

## 2. Experimental

Pure shear testpieces were used, shown in Figure 1, for the measurements on SBR, with a length of 180 mm, a width of 34 mm and a central thickness of about 0.75 mm. They were bonded to long metal end-pieces, one of which was held fixed and the other was fastened to a moveable clamp. An initial cut was made along the center line of the specimen from one end, about 40 mm long. The moveable end-piece was then pulled away from the stationary one, either to a fixed distance, putting the specimen under a pre-determined pure shear strain, or cycled between the unstrained position and a pre-determined position, subjecting the specimen to an intermittent pure shear strain. In the first case the specimen was under a constant pure shear strain and in the second it was strained intermittently, at a frequency of 1.8 Hz.

For these samples the tearing energy  $G$  is given by (1,5)

$$G = W'/t_0 \quad (4)$$

where  $t_0$  is the thickness of the central part of the test specimen and  $W'$  is the amount of elastic energy stored in the specimen per unit length. Values of  $W'$  were determined from experimental

relations between applied load and displacement for each specimen. Because Equation 4 does not contain the length  $c$  of the crack, the tear energy, and hence the rate of growth of a crack, is in principle constant for pure shear test specimens. This is a considerable experimental advantage.

Rates of crack growth were measured at various values of the average applied strain  $\epsilon$ , given by the ratio  $d/h_0$  of displacement  $d$  to unstrained width  $h_0$ . The test chamber could be evacuated to a low pressure of air by means of a vacuum pump, and heated by radiant heaters.

Measurements on a BR vulcanizate were carried out by propagating a tear through a thin strip, about 1 mm thick, at a constant speed, and measuring the average tearing force  $F$ . In this case the tearing energy  $G$  is given by (1)

$$G = 2F/t \quad (5)$$

where  $t$  is the width of the torn path, measured after tearing. The value of  $t$  was generally 50% larger than the thickness of the sheet because the tear tended to run at an angle of about  $45^\circ$  to the plane of the sheet (6).

The compounds used are given in Table 1. For SBR they were chosen to give: SBR-1, C-C crosslinks; SBR-2, mainly monosulfidic crosslinks; and SBR-3, mainly polysulfidic crosslinks. Crosslinking was effected by heating for 1 h at  $150^\circ\text{C}$ . For BR a standard sulfur recipe was used, crosslinked for 50 min at  $140^\circ\text{C}$ .

### 3. Results and discussion

#### (a) The effect of air pressure for SBR

Various air pressures were used, in the range: 0.01 mm Hg to 760 mm Hg (one atmos.). Results for SBR-2 are shown in Figures 2 and 3. Over a wide range of values of tearing energy  $G$  the rates of crack growth under steady and intermittent loading were in good agreement with Equations 2 and 3, with values of the exponents  $\alpha$  and  $\alpha'$  of 4.3 and 3.4, respectively. In all cases they were lower at low air pressures, by a constant factor, independent of the tear energy. Thus, removing air completely has the same proportionate effect on slow and fast rates of crack growth, reducing them to about 30% of the values at one atmosphere of air in the case of intermittent loading and to about 25% in the case of steady loading.

There was some indication that the effect of air pressure disappeared at extremely low air pressures, below about 1 mm Hg, for intermittent loading, Figure 5, but this feature was not apparent in the measurements under continuous loading, Figure 4. In any case, the major changes occurred between 0.001 and 1 atmos, and over this range the linear relations shown in Figures 4 and 5 have the same slope, about 0.12. Thus, the effect of atmospheric pressure appears to be independent of the level of stress or whether it is intermittent or continuously applied. It can be described by the empirical relations:

$$dc/dt = AG^{\alpha} (P/P_0)^{\beta} \quad (6)$$



and

$$dc/dn = A' G^{\alpha'} (P/P_0)^{\beta} \quad (7)$$

over the pressure range from  $P = 0.001$  atmos up to the reference pressure  $P_0$  ( $= 1$  atmos), where  $\beta = 0.12$  for compound 2.

Experiments with SBR-1 and SBR-3 under continuous loading yielded values for the exponent  $\beta$  of 0.082 and 0.185, respectively. Thus, the compound with C-C crosslinks is the least affected by the atmospheric pressure, the one with mainly monosulfidic crosslinks is more strongly affected and the compound with mainly polysulfidic crosslinks is the most affected. These observations are consistent with the known susceptibility of these materials to oxidation.

#### (b) Effect of temperature for SBR

Values of the rate of crack growth  $dc/dt$  for SBR-3 under steady tearing conditions were determined over the temperature range:  $25^{\circ}\text{C}$  to  $100^{\circ}\text{C}$ . The results are plotted in Figures 6 and 7 on logarithmic scales against the reciprocal of the test temperature  $T$ . The results shown in Figure 6 were obtained at an air pressure of one atmos; those in Figure 7 were obtained at a reduced pressure of 0.1 mm Hg. In both cases they show a strong dependence on temperature, the relations being approximately parallel for different values of the imposed average strain at  $25^{\circ}\text{C}$ . Thus, the rate of crack growth appears to depend exponentially upon the reciprocal of temperature, with an apparent activation energy  $E$

which is independent of applied stress:

$$dc/dt = AG^{\alpha}(P/P_0)^{\beta} \exp(-E/R)[(1/T) - (1/T_0)] \quad (8)$$

where the reference temperature  $T_0$  is ambient temperature (298°K).

It should be noted that these measurements were made using a fixed displacement of the specimen clamps. Because a significant amount of thermal expansion occurred when the temperature was raised, the strain energy stored in the specimen at a given displacement became smaller and the amount of energy  $G$  available for tearing was correspondingly reduced. However, experimentally-determined relations between stress (and strain energy  $G$  available for tearing) and the average strain measured at that temperature were found to be substantially the same over the temperature range 20°C to 80°C. Values of average strain, and hence  $G$ , were therefore computed from the observed values of the unstretched width  $h_0$  at each temperature. They were employed to calculate best-fit constants in Equation 8. Values obtained in this way for SBR-3 are given in Table 2. Relations corresponding to Equation 8 with these values of the constants are represented by the full curves of Figures 6 and 7. They are seen to describe the experimental results with reasonable accuracy.

Experiments were also carried out at various temperatures with the clamp displacements chosen so as to give constant values for tearing energy, i.e., making allowance for thermal expansion of the test specimen. Measured values of the rate of crack growth are

plotted in Figure 8 against the reciprocal of the test temperature, for two different values of tearing energy  $G$ . The full lines in Figure 8 were calculated from Equation 8, using the fitting constants given in Table 2. They are seen to describe the experimental results well. Furthermore, there is no significant difference in the values of the fitting constants at 1 atmos and at 0.1 mm Hg. Thus, it is clear that Equation 8 represents the rate of crack growth of this vulcanizate over a wide range of applied loads, test temperatures and air pressures. The effects of these variables are apparently independent and multiplicative, to a first approximation.

Similar measurements of the rate of steady tearing were made for SBR-1. The results are shown in Figures 9 and 10. A value for activation energy  $E$  of  $25 \pm 2$  kcal/mole was obtained from them, substantially less than the value of  $32 \pm 3$  kcal/mole deduced for SBR-3 but considerably higher than would be expected from the WLF relation that accounts for changes in viscous energy losses with temperature. The effective activation energy from the WLF rate-temperature equivalence would be about 17 kcal/mole for SBR (with a glass transition temperature of  $-55^{\circ}\text{C}$ ) over the present temperature range.

Resistance to tearing has been attributed mainly to internal energy dissipation as the material is deformed to the point of rupture (7). Evidence for this is the good correlation obtained between tear energy  $G$  and the value of the out-of phase shear modulus  $G''$ , a measure of internal dissipation (7). Moreover,

provided care is taken to prevent the tear becoming rougher at lower temperatures or higher rates of tearing, experimental relations between tear energy and tear rate are found to follow temperature shifts in good agreement with the predictions of the WLF relation for segmental mobility as a function of temperature (7,8). Similarly, the tensile strength and extensibility of a simple SBR vulcanizate depend upon rate of stretching and temperature as expected for viscous processes (9).

But these studies of tearing and tensile strength have been largely carried out at low temperatures, near the glass temperature  $T_g$ , and at temperatures up to about  $100^{\circ}\text{C}$  above  $T_g$ , where changes in molecular mobility and internal energy dissipation are most pronounced. For example, tear strength results reported previously for an SBR vulcanizate were found to be in good accord with the predictions of the WLF relation over a wide temperature range, from  $-40$  to  $+100^{\circ}\text{C}$  (8). On closer inspection, however, the results at the higher temperatures,  $25^{\circ}$  to  $100^{\circ}\text{C}$ , are in better agreement with an activation energy of 27 to 32 kcal/mole, as in the present measurements, rather than with the lower value, about 17 kcal/mole expected from the WLF relation.

Thus, there is reason to question the applicability of the WLF rate-temperature equivalence to tear strength results at high temperatures. In an attempt to clarify this point, measurements have also been carried out on a BR vulcanizate, with a much lower glass transition temperature,  $-96^{\circ}\text{C}$ . The equivalent activation energy for viscous processes at temperatures in the range 25 to

130°C is obtained from the WLF relation as only about 10 kcal/mole in this case. Only small effects of temperature on the tear strength would therefore be expected. The experimental results are described in the following section.

(c) Effect of temperature for a BR vulcanizate

Measured tear strengths are plotted against the rate of tearing in Figure 11, using logarithmic scales for both axes. Although the results do not fall on strictly parallel curves, it is possible to obtain shift factors  $\log a_T$  by superimposing them as closely as possible at a given value of tear energy  $G$ . The results are plotted in Figure 12 as a function of the reciprocal of absolute temperature, in order to obtain an effective activation energy from the slope. A value was obtained in this way of about 18-20 kcal/mole, much larger than expected for a purely viscous process over this temperature range. Thus, as found for the SBR vulcanizates, there is a marked discrepancy between the observed temperature dependence of tear energy and that predicted by the WLF relation.

We are therefore led to conclude either that the WLF relation seriously underestimates the changes in viscous energy losses with temperature at high temperatures, or that there is an additional temperature-dependent part of the tear strength that does not obey the WLF relationship. The latter alternative seems more likely. A possible origin of the second term in tear strength is discussed in the following section.

(d) Possible temperature dependence of  $\underline{G}_0$

The tear energy  $\underline{G}$  can be expressed as the sum of two terms, an inherent, "threshold" strength  $\underline{G}_0$  that exists in the absence of any dissipative process, and a term  $\underline{H}\underline{G}_0$  due to internal energy losses that are generated in deforming the sample to the point of rupture (10):

$$G = G_0 (1 + H) \quad (9)$$

where  $\underline{H}$  is generally much greater than unity. The factor  $\underline{H}$ , and hence the term in parentheses, is expected to follow the WLF rate-temperature equivalence for simple visco-elastic solids. We now consider a separate temperature dependence of the threshold strength  $\underline{G}_0$ , independent of the rate of tearing.

By subtracting rate-temperature shifts  $\log a_T$  calculated from the universal form of the WLF relation, from the experimentally-observed ones, and applying a shift in  $\log \underline{G}$ , denoted  $\Delta \log \underline{G}_0$ , to account for the discrepancy, Figure 13, values were obtained for  $\Delta \log \underline{G}_0$  that depend on temperature as shown in Figure 14. They decreased markedly with temperature, from 2X - 5X at 25°C down to 1X at the reference temperature of 130°C. They are rather similar in general form for the different materials, even though the overall dependences of tear energy upon temperature were quite different.

Previous measurements of the threshold tear energy  $G_0$ , carried out mainly at high temperatures, have given values of about 50 J/m<sup>2</sup>. If the inferred changes in  $G_0$  are applied to this value, assumed to hold at 130°C, they yield a dependence of  $G_0$  on temperature from 100 - 250 J/m<sup>2</sup> at 25°C to 50 J/m<sup>2</sup> at 130°C. This decrease may reflect the thermal sensitivity of sulfur crosslinks (11,12).

For vulcanizates of both polymers the anomalously strong dependence of tear energy upon temperature at high temperatures can be accounted for as a sum of two effects: a viscous-loss contribution that decreases with temperature in accordance with the WLF relation, and a decrease in the "intrinsic" strength of the molecular network.

But it should be pointed out that the magnitudes estimated here for  $G_{0,T}$  (Figure 14) are quite approximate. They are based on relatively imprecise measurements of tear strength over limited ranges of rate and temperature, that are then compared with extrapolations of the WLF relation. Nevertheless, the values obtained seem inherently plausible.

They lead to an important conclusion, that the threshold strength itself will become large at low temperatures. Relatively few direct measurements have been made of threshold strength, and they have usually been carried out at high temperatures, to approach non-dissipative conditions. Thus, no direct evidence is known to the present authors of a significant temperature

dependence of  $G_0$ . However, if it is, indeed, the case that  $G_0$  increases as the temperature is lowered, then the observed tear strength will be expected to rise rapidly (as is observed) because the effect is coupled with a simultaneous rise in dissipative processes, Equation 9.

Moreover, different temperature dependences will be expected for different polymers. Similarly, the effect of temperature on tear strength will presumably be different for materials with different crosslinking systems, even when viscous effects are the same. Further experimental studies are needed in this area.

#### 4. Conclusions

1. A linear relationship has been found to hold for SBR vulcanizates between the logarithm of the rate of steady tearing and the logarithm of the air pressure, over the range 0.1 mm Hg to 760 mm Hg. The slope varied from 0.08 for a C-C crosslinked material to 0.18 for a polysulfide-crosslinked material. Similar effects of air pressure were found in the rate of crack propagation under intermittent loading. They are attributed to attack of oxygen (in air) on highly stressed bonds, either sulfidic or hydrocarbon.

2. Approximately linear relationships were found to hold between the logarithm of the rate of steady tearing and the reciprocal of the test temperature for temperatures between 25°C and 130°C. Apparent activation energies were deduced from the slopes, of 32



kcal/mole for a polysulfide-crosslinked SBR compound, 25 kcal/mole for a C-C crosslinked SBR compound, and 20 kcal/mole for a sulfur vulcanizate of BR. For SBR, these values were independent of the air pressure.

3. These activation energies are far too large to be accounted for by changes in segmental mobility (and hence in internal energy dissipation) with temperature, for simple visco-elastic materials at temperatures far above  $T_g$ . The discrepancies are attributed to a second temperature-dependent factor. It is suggested that the threshold tear strength  $G_0$ , i.e., the strength under non-dissipative conditions, is itself temperature dependent.

4. Estimates of the changes in  $G_0$  with temperature have been obtained from the observed discrepancies in temperature dependence of tear strength. They correspond to a decrease from about 100 - 250 J/m<sup>2</sup> at 25°C to about 50 J/m<sup>2</sup> at 130°C. These changes in  $G_0$  may reflect the stress-activated rupture of thermally-sensitive bonds.

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Table 1. Compound formulations.

	SBR-1	SBR-2	SBR-3	BR
SBR 1502	100	100	100	-
Diene 55	-	-	-	100
Stearic Acid	-	2	2	2
Zinc Oxide	-	5	5	5
Sulfur	-	-	2	2
SANTOCURE <sup>(a)</sup>	-	-	-	1
MOR <sup>(b)</sup>	-	-	1	-
TMTD <sup>(c)</sup>	-	4	-	-
DCP <sup>(d)</sup>	0.5	-	-	-

(a) N-cyclohexyl-2-benzothiazolesulfenamide.

(b) 2-(Morpholiniothio)benzothiazole.

(c) Tetra methylthiuramdisulfide.

(d) Dicumyl peroxide.

Table 2. Fitting constants in Equation 8.

	log A at 25°C ( SI units )	$\alpha$	E (kcal/mole)
SBR-1			
1 atm.	-14.6	3.5±0.5	25±2
0.1 mmHg	-14.5	3.6±0.5	25±2
SBR-3			
1 atm.	-18.9	4.4±0.5	32±3
0.1 mmHg	-18.6	4.4±0.5	32±3
BR			
1 atm.	-25.1	7.3±0.7	19±1

### Figure Legends

1. Test specimen
2. Experimental relations between the rate  $\frac{dc}{dt}$  of steady tearing and tear energy  $G$  for SBR-2 at different air pressures.
3. Experimental relations between the rate  $\frac{dc}{dn}$  of intermittent tearing and tear energy  $G$  for SBR-2 at different air pressures.
4. Experimental relations between the rate  $\frac{dc}{dt}$  of steady tearing and air pressure  $P$  for SBR-2 at various tear energies  $G$ .
5. Experimental relations between the rate  $\frac{dc}{dn}$  of intermittent tearing and air pressure  $P$  for SBR-2 at various tear energies  $G$ .
6. Effect of temperature on the rate  $\frac{dc}{dt}$  of steady tearing of SBR-3 at 1 atmos air pressure.
7. Effect of temperature on the rate  $\frac{dc}{dt}$  of steady tearing of SBR-3 at 0.1 mm Hg air pressure.
8. Effect of temperature on the rate  $\frac{dc}{dt}$  of steady tearing of SBR-3 at constant tear energy  $G$ .
9. Effect of temperature on the rate  $\frac{dc}{dt}$  of steady tearing of SBR-1 at 1 atmos air pressure.
10. Effect of temperature on the rate  $\frac{dc}{dt}$  of steady tearing of SBR-1 at 0.1 mm Hg air pressure.
11. Effect of temperature on the tear strength  $G$  of a BR compound, torn at various rates.
12. Shift factors  $\log a_T$ , from the results shown in Figure 11, plotted against the reciprocal of temperature.

13. Method of determining  $\Delta \log G_0$  from the experimental relations shown in Figure 11.

14. Dependence of  $\Delta G_0$  upon temperature for SBR-1, SBR-3 and BR vulcanizates.

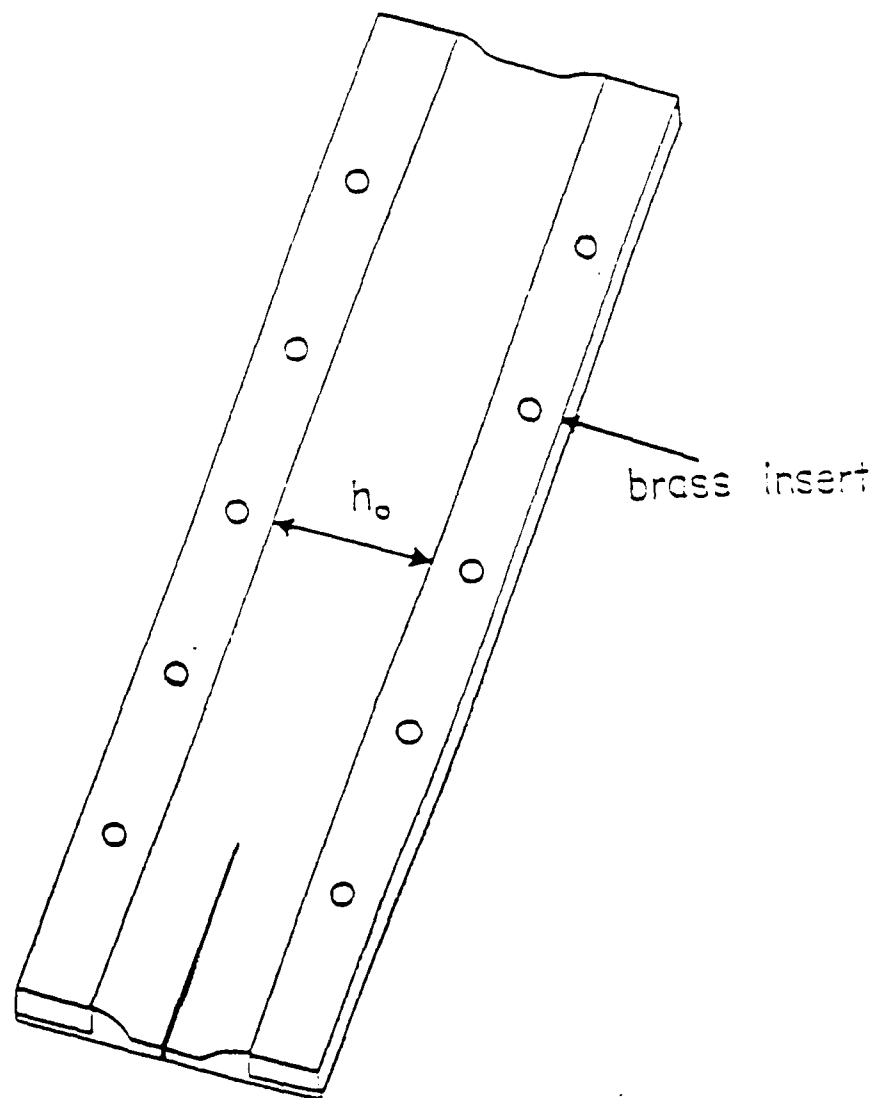


FIGURE 1

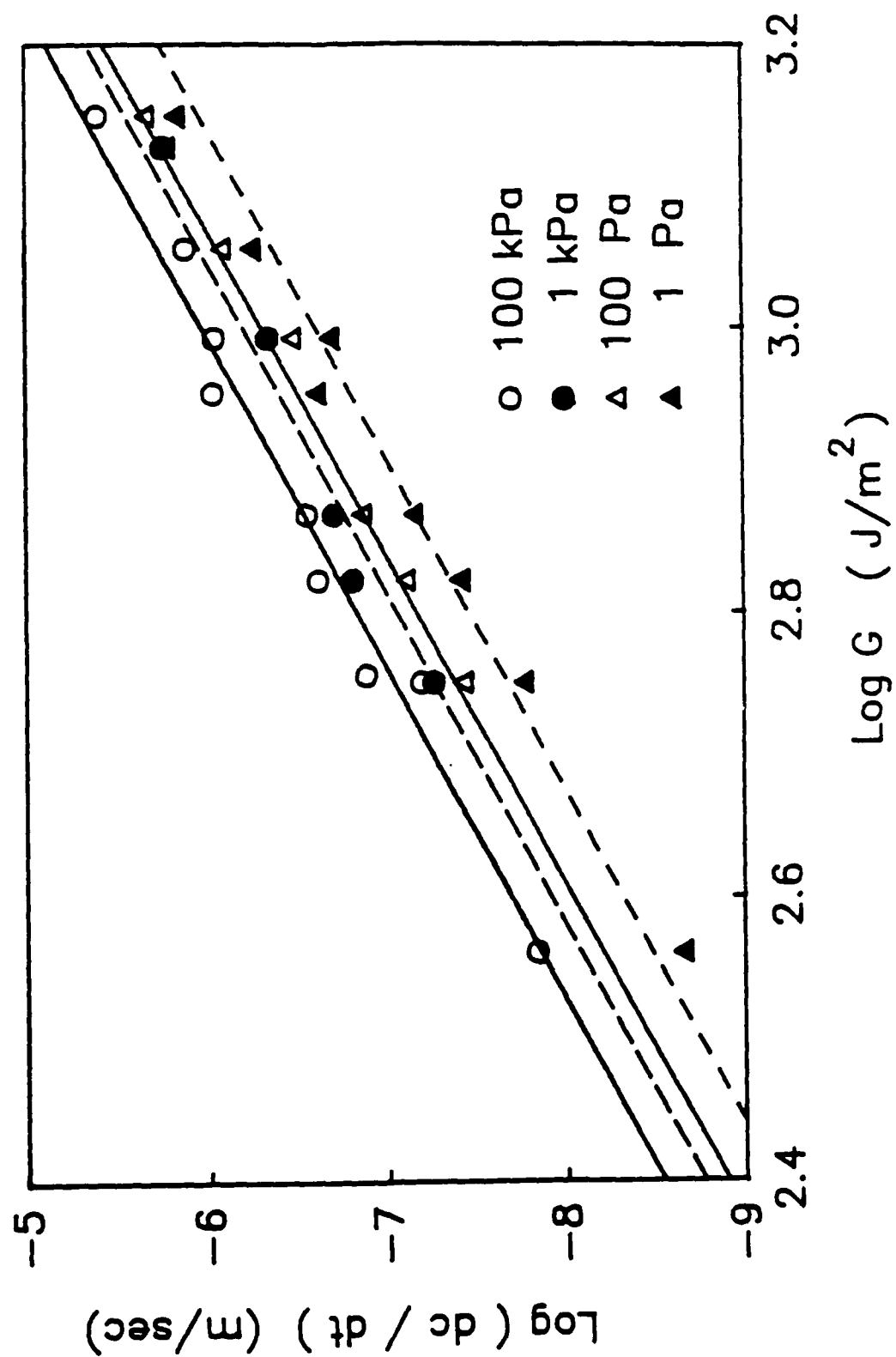


FIGURE 2



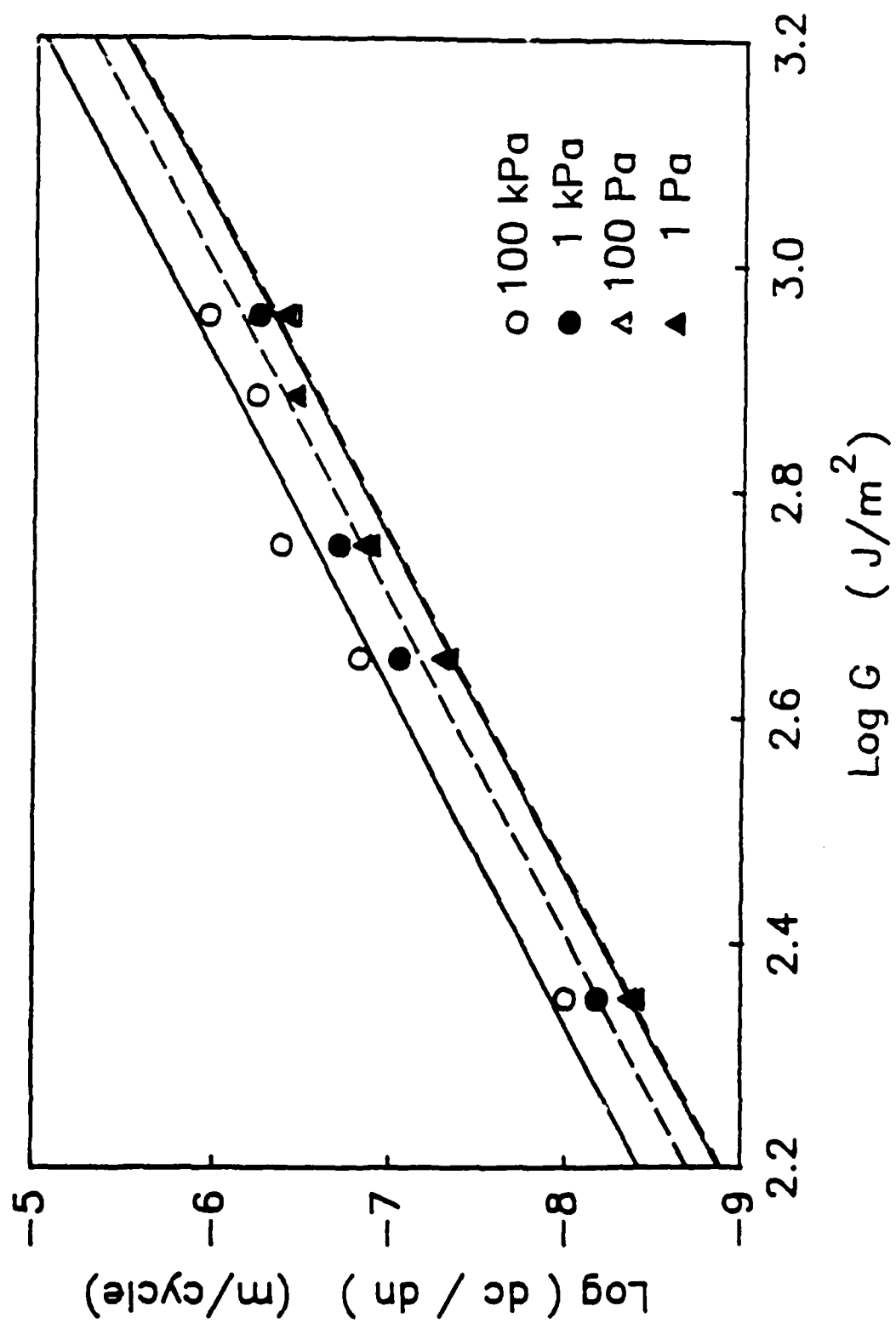


FIGURE 3

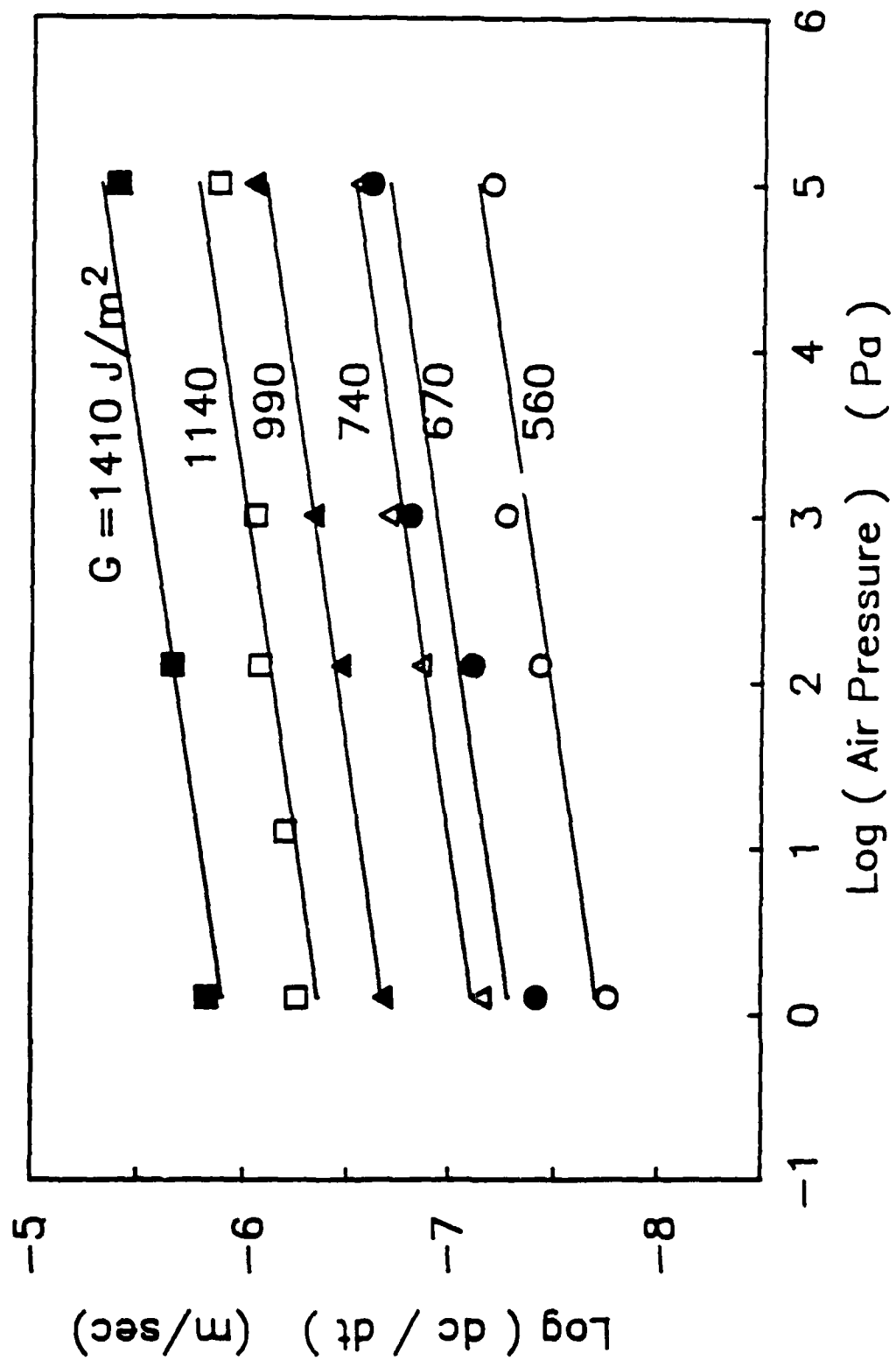


FIGURE 4

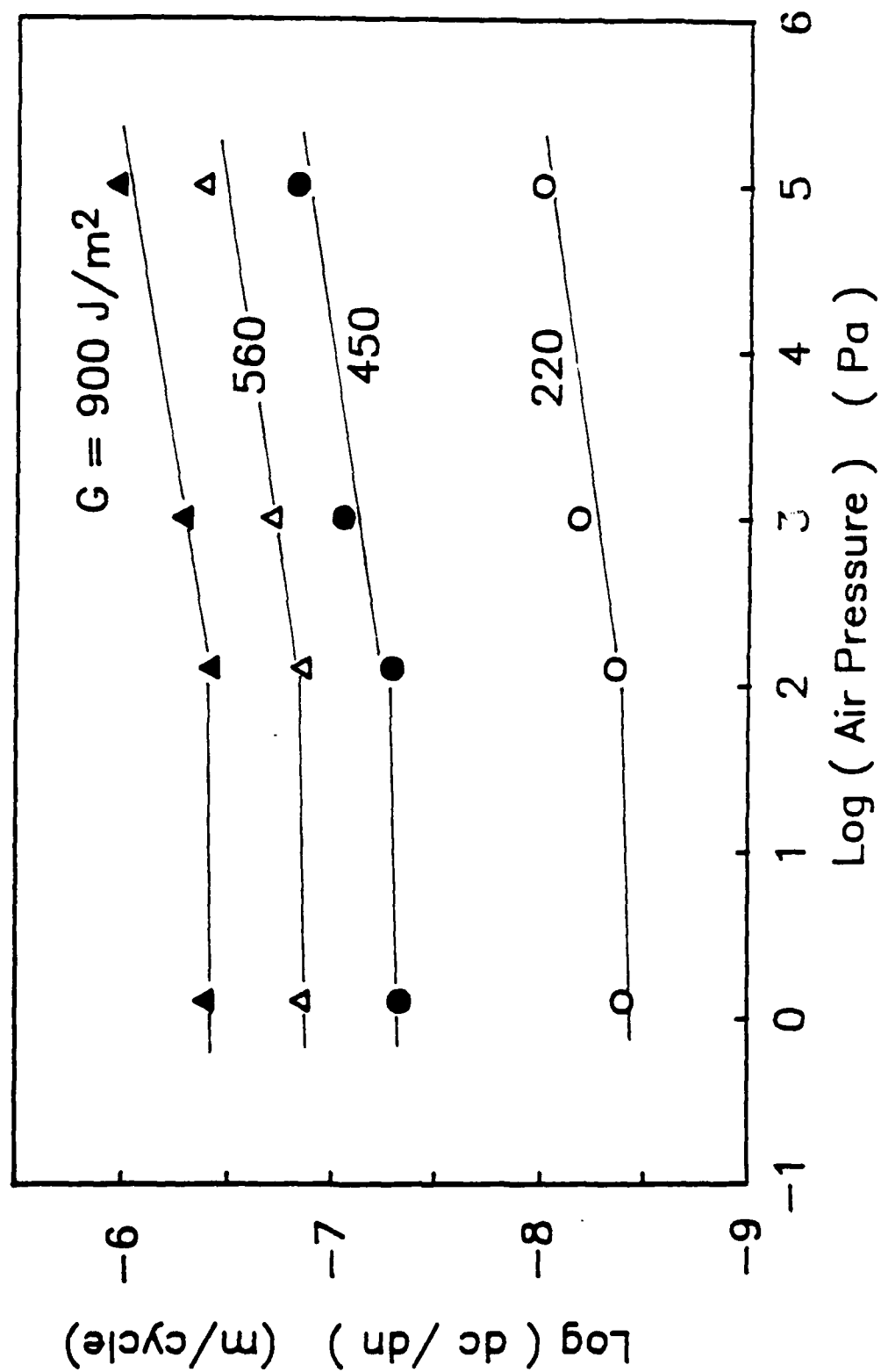


FIGURE 5

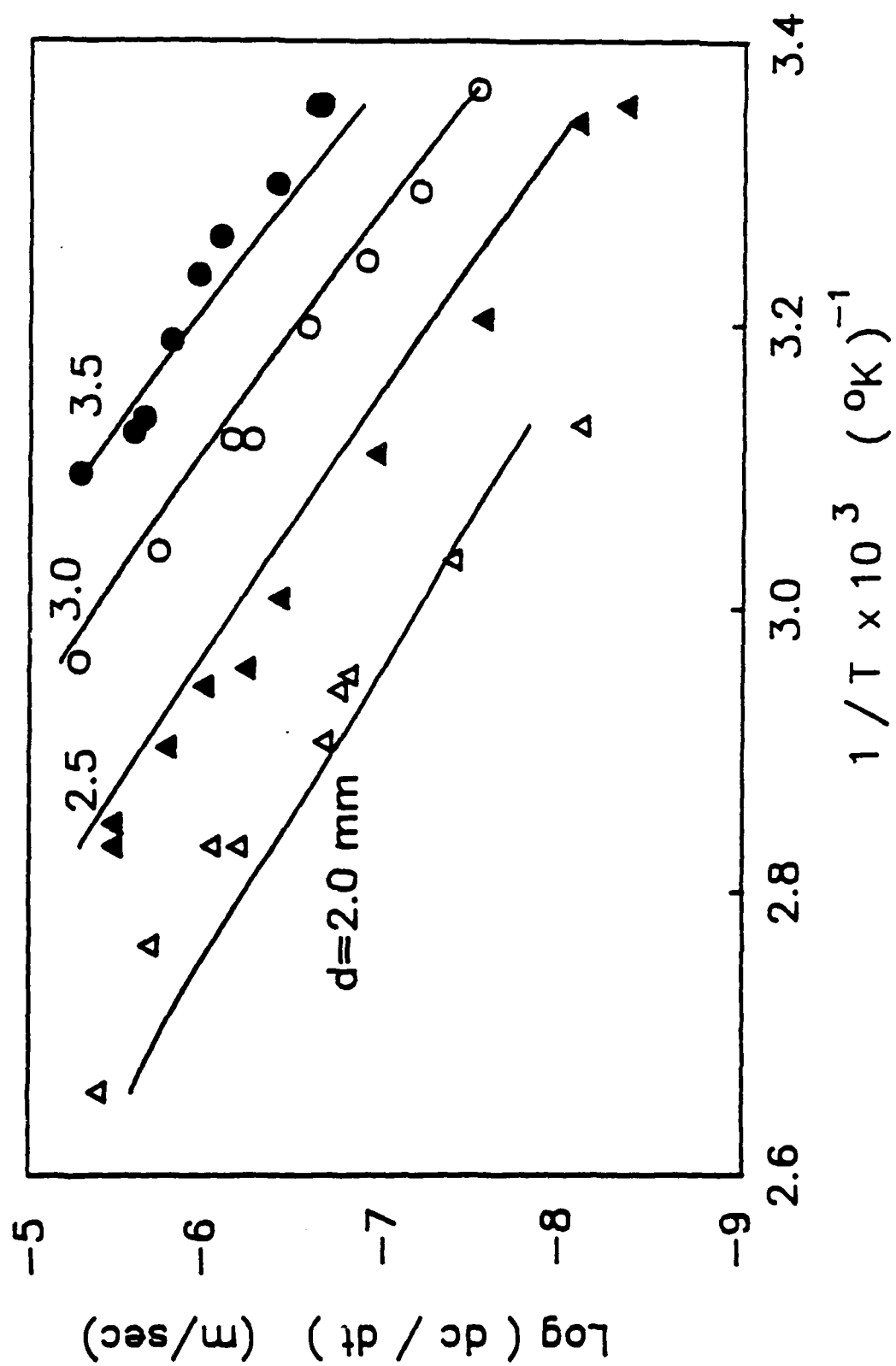


FIGURE 6

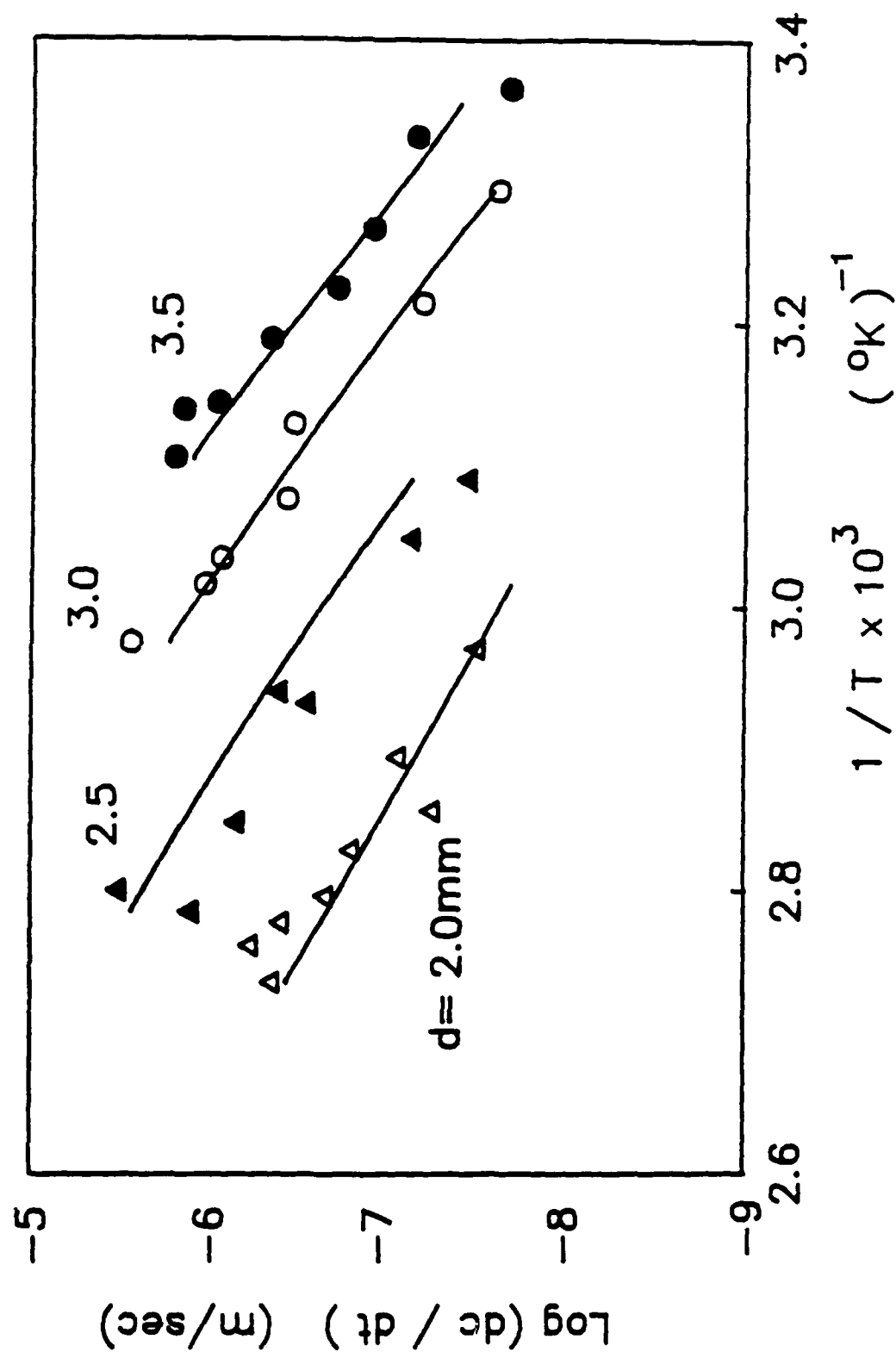


FIGURE 7

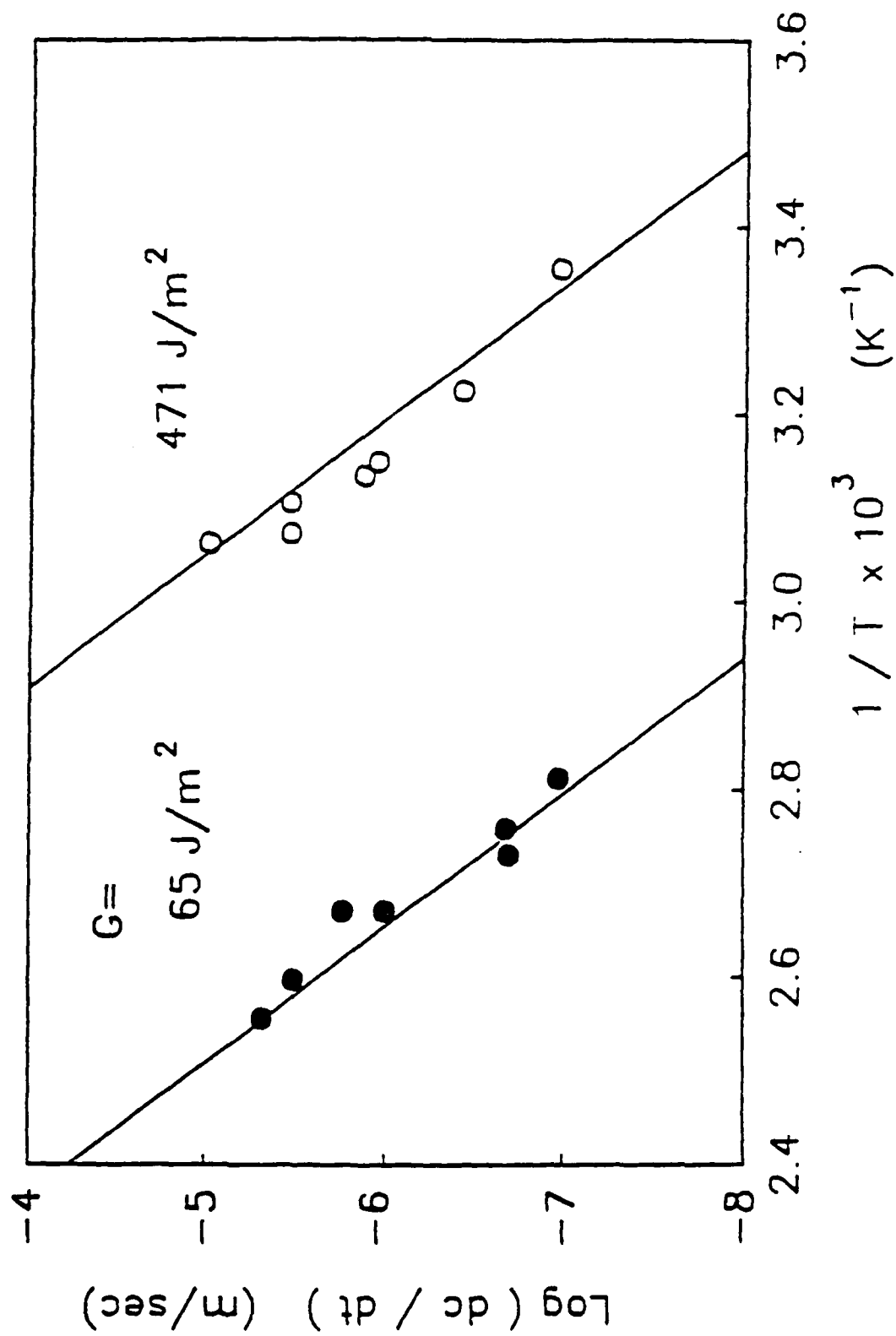


FIGURE 8

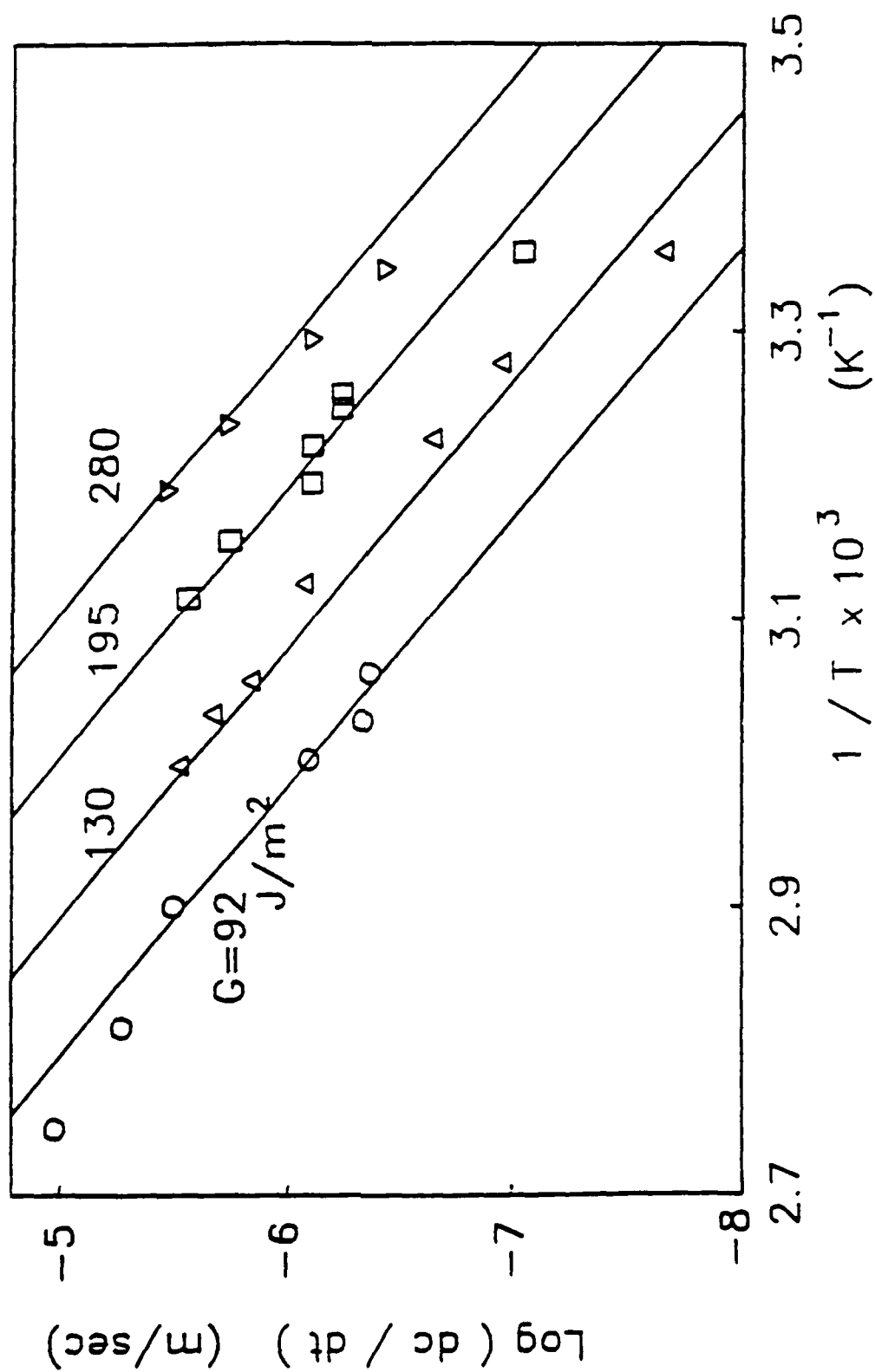


FIGURE 9

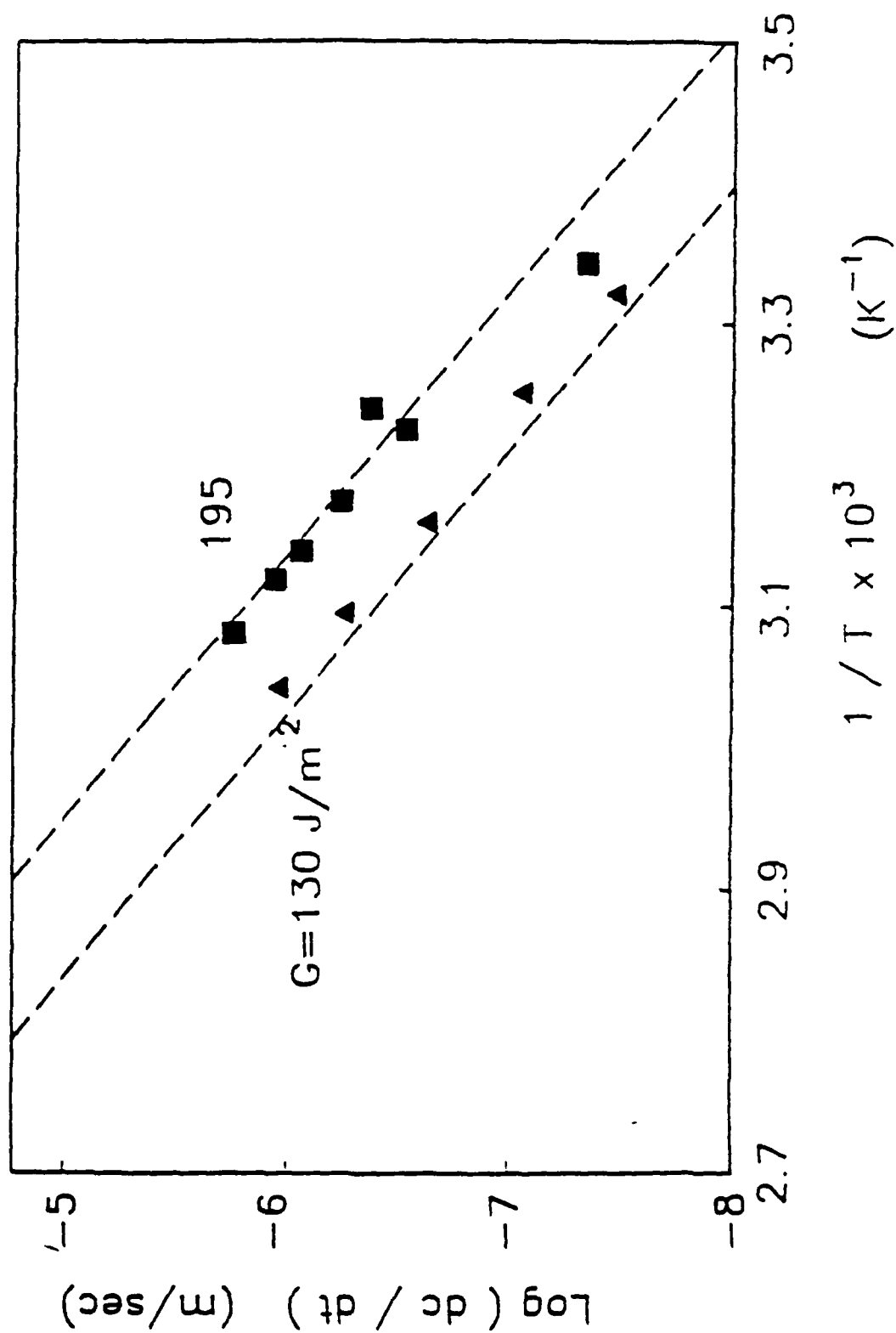


FIGURE 10



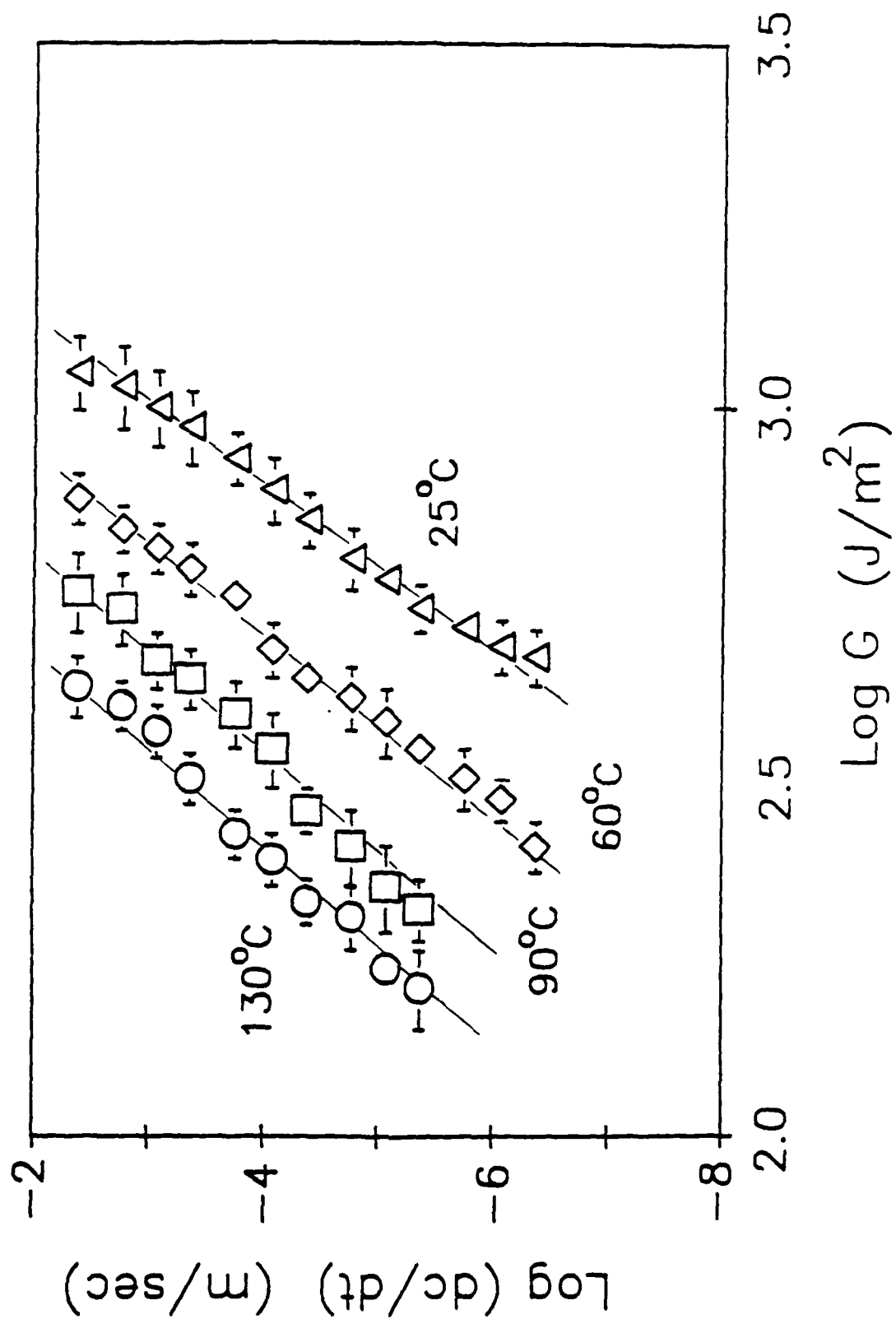


FIGURE 11

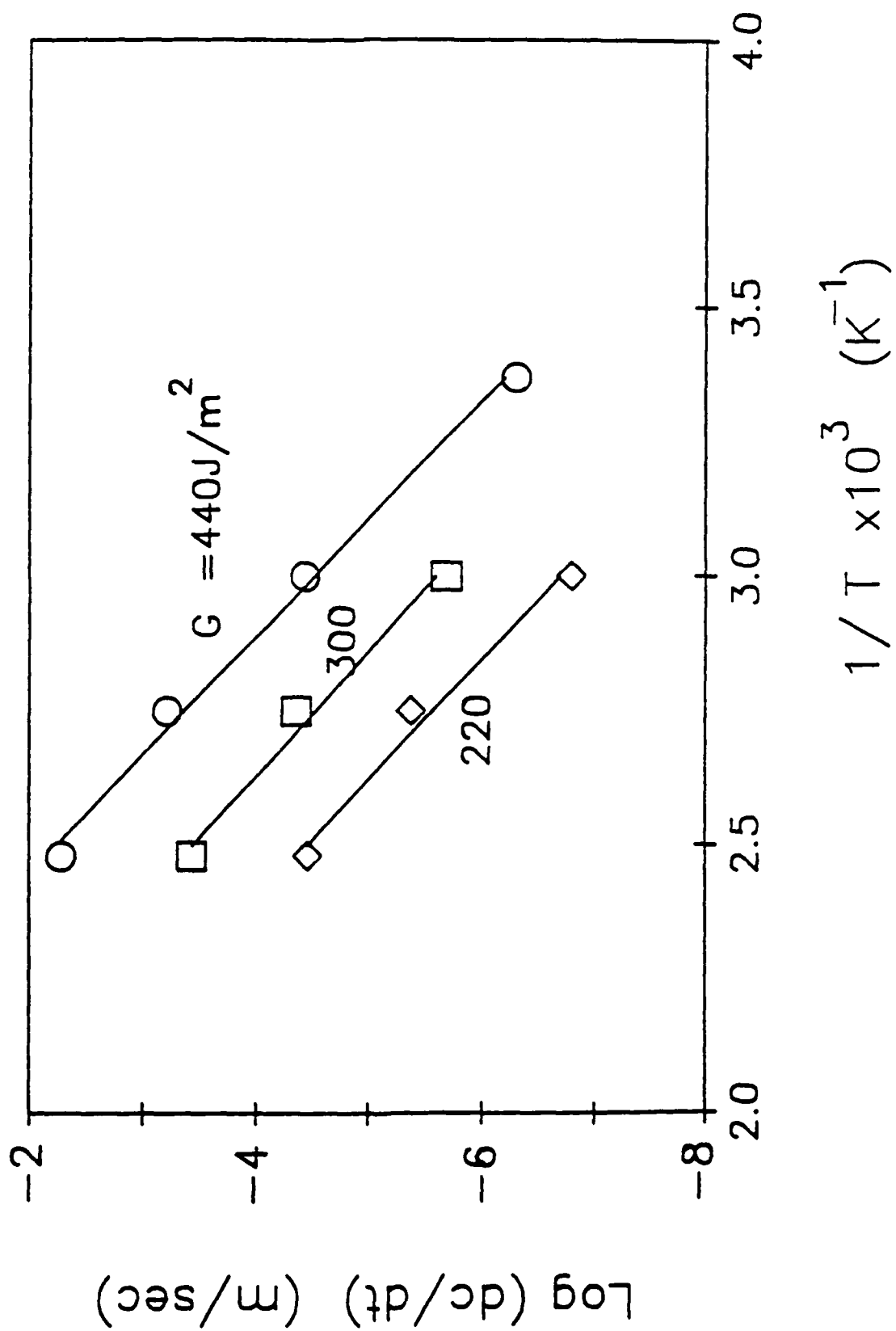


FIGURE 12

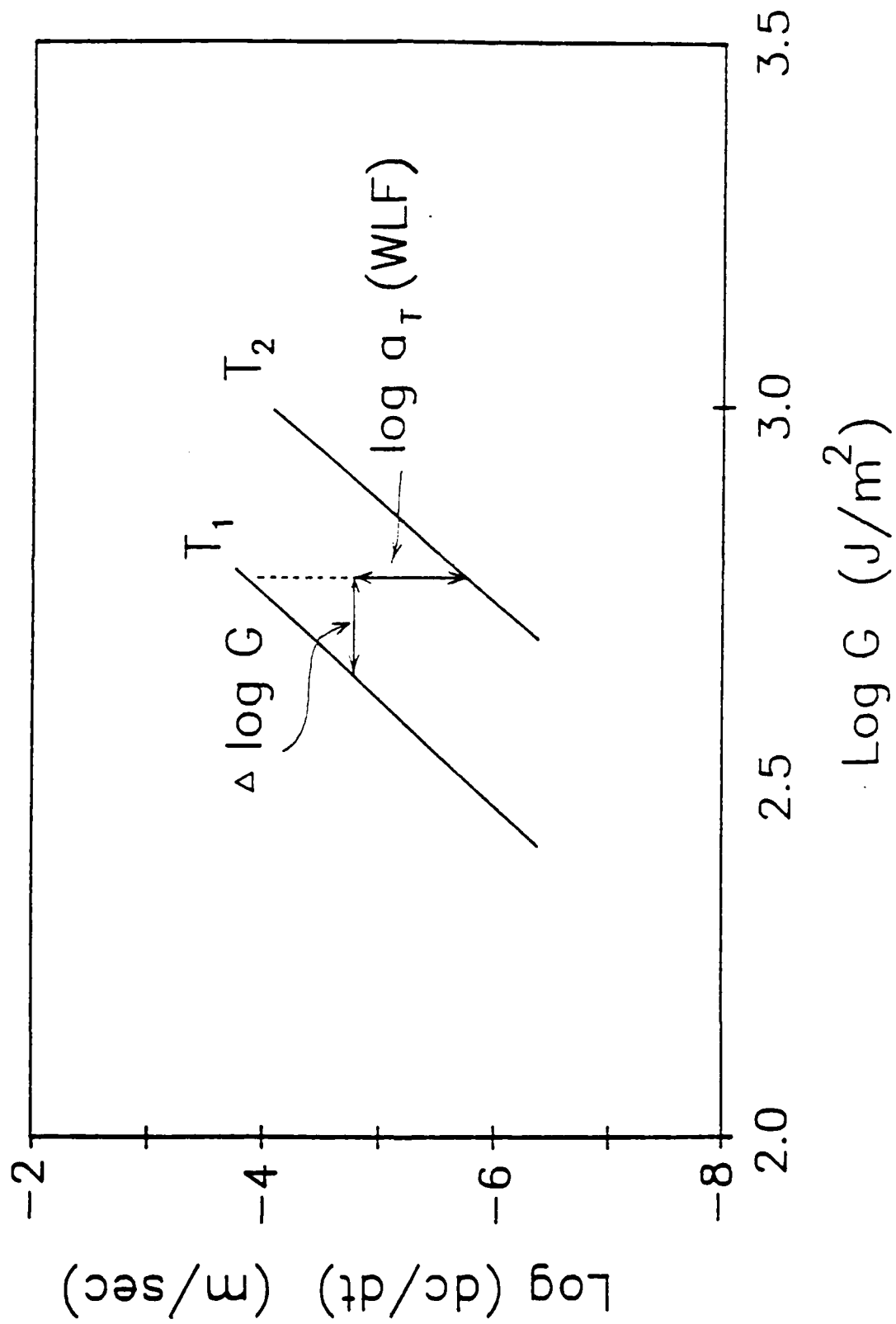


FIGURE 13

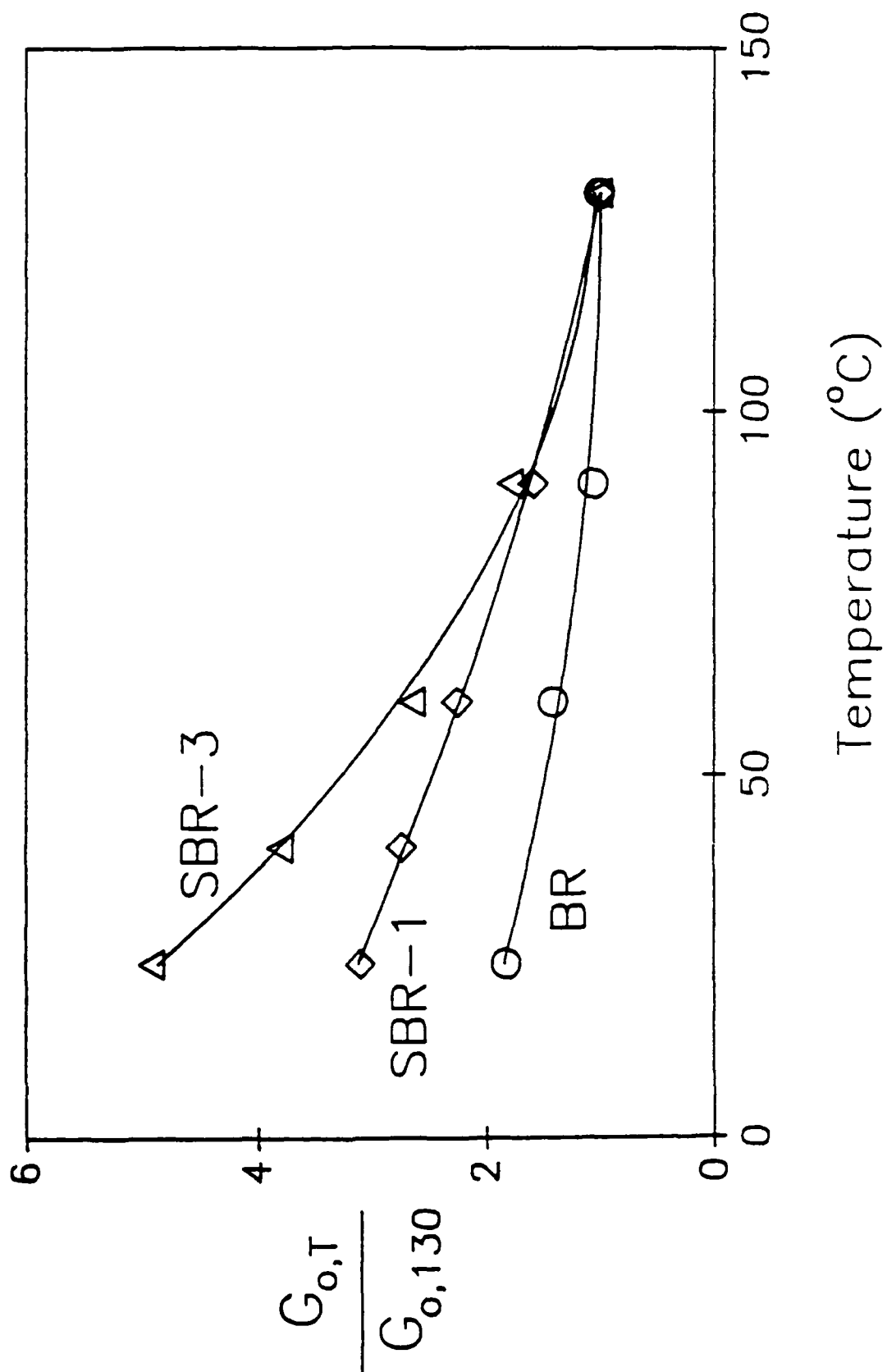


FIGURE 14

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